A DEVELOPMENT STUDY OF MICROALLOYED STEEL (HSLA) THROUGH EXPERIMENTAL EXPLORATION

A THESIS SUBMITTED IN PARTIAL FULFILLMENT OF THE REQUIREMENTS FOR THE DEGREE OF

Bachelor of Technology

In

Metallurgical and Materials Engineering

By

ARINDAM SARKAR (108MM051)

SWAYAMBHU PANDA (108MM052)

Under the guidance of

Prof.B.C.Ray

Department of Metallurgical and Materials Engineering

National Institute of Technology

Rourkela

2012

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CERTIFICATE

This is to certify that the thesis entitled "**A DEVELOPMENT STUDY OF MICROALLOYED STEEL (HSLA) THROUGH EXPERIMENTAL EXPLORATION**" submitted by **Arindam Sarkar (108MM051)** and **Swayambhu Panda (108MM052)** in partial fulfilment of the requirements for the award of **Bachelor of Technology Degree** in **Metallurgical and Materials Engineering** at **National Institute of Technology, Rourkela** is an authentic work carried out by them under my supervision and guidance.

To the best of my knowledge the matter embodied in the thesis has not been submitted to any other University/Institute for the award of any Degree or Diploma.

Date: 9/5/12

9/5/12

Prof. B.C.Ray

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ABSTRACT

HSLA steel is indispensable to the development of modern and future civilisations. The selection of optimum solutionizing temperature range for microalloyed steel grades is of utmost importance as it can significantly reduce energy consumption. Reheating studies were carried out on two different micro-alloyed steel grades (strip samples) containing Niobium. The reheating temperature range and the effect of niobium content on the final properties of the steel were established by performing a series of quench and temper experiments, followed by hardness testing. A different set of samples (of the same grades) were subjected to heating in a controlled atmosphere (Nitrogen atmosphere) to mark the differences as compared to heating in open (air) atmosphere. Hardness variation across the strip width was also tested. Microstructural analysis was done using SEM and EDX for further investigation of the results obtained. For both grades (and for both cases of heating in open and nitrogen atmosphere), hardness increased with an increase in solutionizing temperature. Also, at a particular solutionizing temperature, the hardness value was higher for the grade containing greater niobium content. This effect can be attributed to the increasing fraction of niobium that dissolves into the solution (matrix) with increasing solutionizing temperature. However, after a particular temperature range, increasing the solutionizing temperature does not significantly increase the hardness, indicating the saturation of Nb levels in the solution.

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CHAPTER 1

INTRODUCTION

1. INTRODUCTION

1.1 HSLA STEEL: SALIENT FEATURES

Microalloyed steels, often referred to as High-Strength Low-Alloy Steels (HSLA), are a family of materials strengthened by the elements niobium, vanadium and titanium added either singly or in combination. The microalloying elements are used along with other strengtheners such as boron, molybdenum, chromium, nickel and copper and their use is accompanied by strict control of impurities such as sulphur, oxygen, nitrogen and phosphorus. Strengthening by microalloying dramatically reduces the carbon content which greatly improves weldability and notch toughness.

The single most important microstructural feature of hot-rolled microalloyed steels is ferrite grain size, which lead to both improved strength and notch toughness. A grain size of ASTM 10 (10µ) or finer is the basic building block of these steels which allows the use of other strengthening mechanisms such as solid solution and precipitation hardening all of which lead to a reduction in notch toughness[13][14] .

Micro-alloyed steels, have small additions (< 0.1%) of alloying elements to low carbon steels (0.03%-0.15%C and up to 1.5%Mn) to achieve high yield and tensile strengths.

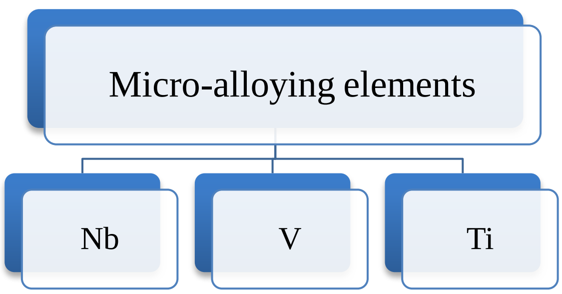

Figure 1.1: Important microalloying elements

Micro-alloying, in terms of chemical composition, creates the pre-requisite for **thermo-mechanical processing** and pursues the following goals: [11]

- ✓ Grain refinement
- ✓ Retarding and/or preventing the recrystallization of hot worked austenite
- ✓ Precipitation strengthening

1.1.1 THERMOMECHANICAL PROCESSING

The science of making HSLA steel was known many years ago, but the traditional rolling routes and heat treatment methods available were not suitable to apply the required process attributes. Hence, extensive research over many decades has led to the development of thermomechanical processing. Thermomechanical treatment is nothing but the metallurgical process which involves the integration of work-hardening and heat treatment methods. The following figure describes the key features of the process:

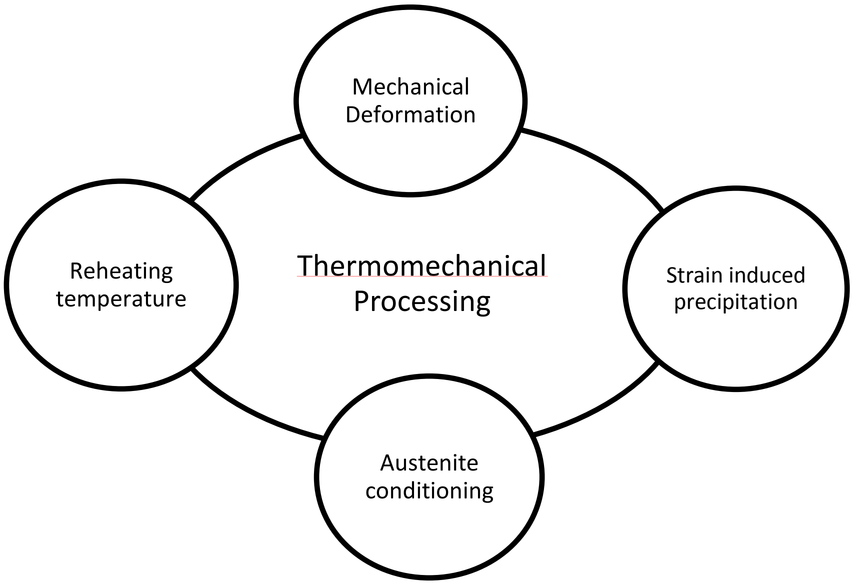

Figure 1.2: Key attributes of thermomechanical processing

For microalloyed steel grades, dissolution during soaking (reheating) and conditioning of austenite (strain, strain rates, deformation temperatures) prior to transformation are extremely important parameters to be taken care of.

In comparison to structural steel grades which are strengthened by solid solution hardening elements, such as Manganese or Silicon and different amounts of pearlite (dependent on carbon content) microalloyed steel grades derive a significant amount of their strength from precipitation hardening by Titanium-Niobium carbonitrides, Vanadium carbides and from the grain refinement caused by thermomechanical treatment. That means during hot rolling, deformation induced precipitation of microalloying elements like Ti(C,N) and Nb(C,N) occurs. These fine precipitates as well as the microalloying elements in solution delay the recrystallization of the deformed austenite which then after the last stand of the finishing mill transforms in a dislocation enriched fine grained ferrite.

The strength level of the HSLA steels can be enhanced both by increasing the amount of precipitates (e.g. higher Ti, V and Nb contents) and by using solid solution hardening elements such as Silicon.

Actually, the austenite grains may recrystallize several times during hot-controlled-rolling, but the total effect of this will be a marked refinement in austenite grains by the time the steel reaches austenite to ferrite transformation temperature. In the later stages of austenite deformation at lower temperatures, recrystallization may not occur. The elongated, flattened (pan-caked) austenite grains may then transform directly to fine ferrite, or may be cooled rapidly from the finish rolling temperature so that austenite to ferrite transformation takes place sub-critically to produce still finer ferrite grains. A schematic diagram of the stages of controlled rolling is shown in figure 1.3.

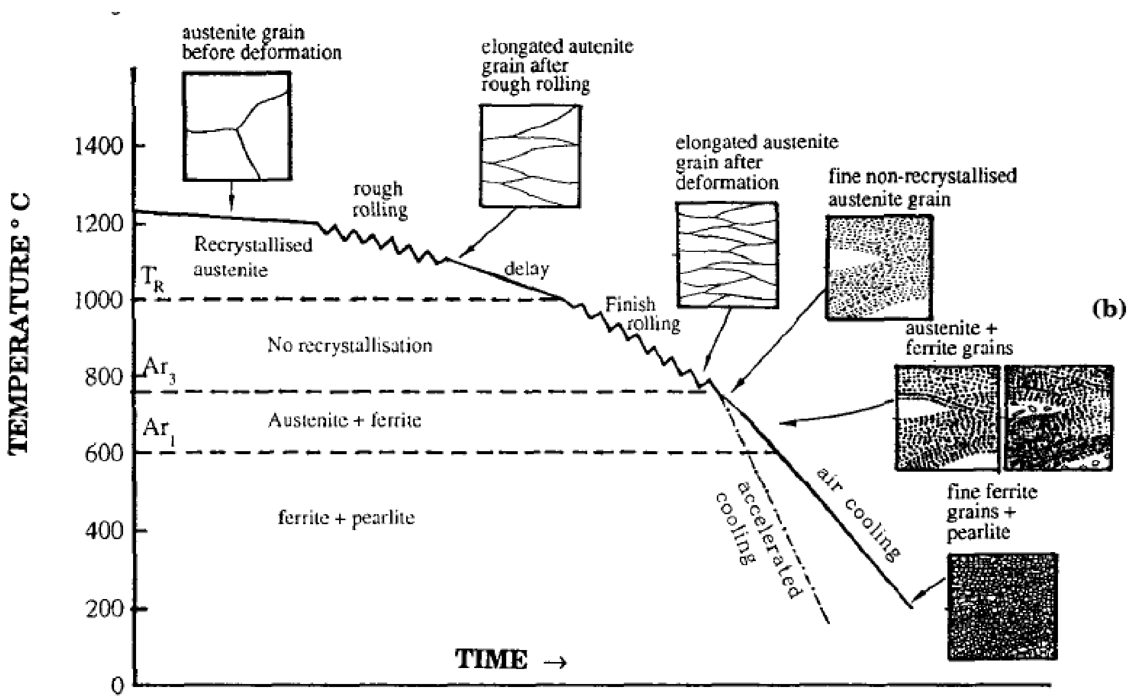

Figure 1.3: Schematic depiction of conventional thermomechanical processing [3]

Ar_3 =Temperature at which austenite to ferrite transformation begins

Ar_1 = Temperature at which austenite to ferrite transformation is complete

T_R = Recrystallization stop temperature

1.1.2 REHEATING TEMPERATURE

The production process of HSLA steel using thermomechanical treatment starts with soaking in a reheating furnace at an appropriate reheating temperature. The reheating temperature thus, is essentially the highest temperature to which the steel is heated during thermomechanical processing, in order to ensure the maximum dissolution of the microalloying elements present. The amount of microalloying content in solution at the end of reheating influences the recrystallization kinetics, recrystallized grain size, grain growth and further precipitation in both austenite and ferrite.

The reheating temperature controls the:

- ✓ initial austenite grain size
- ✓ solution and precipitation of the micro-alloy carbides, nitrides and carbonitrides

1.1.3 ROLE OF NIOBIUM

Niobium is a very important alloying element because it can not only restrain the growth of austenite grain, but also inhibit austenite recrystallization, so controlled rolling and controlled cooling technology is very effective in increasing the strength and toughness of the steels containing niobium [2] .

The effectiveness of niobium in retarding recrystallization of hot worked austenite and controlling grain growth is shown in figure 1.4 and figure 1.5 respectively.

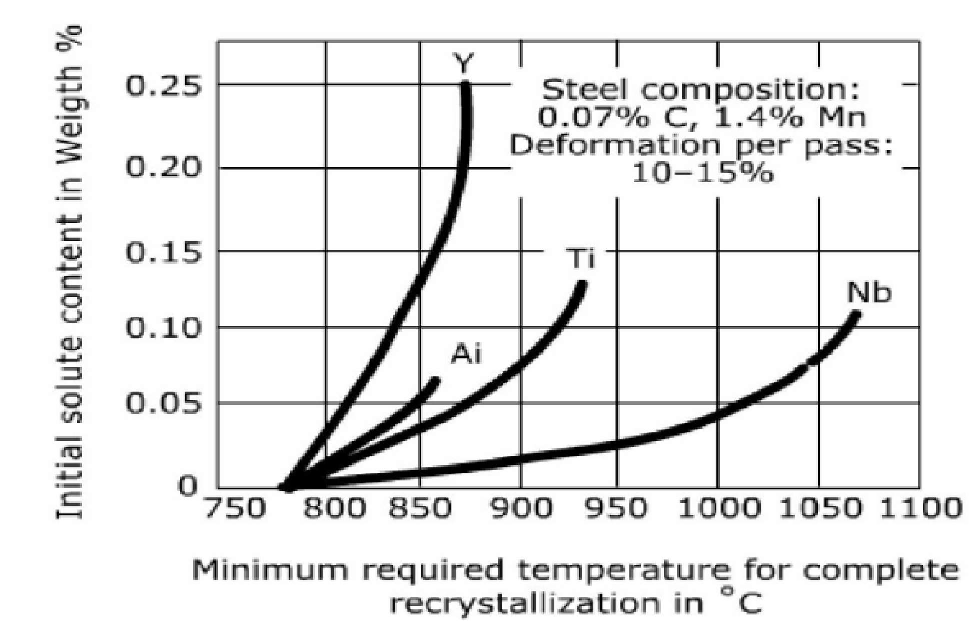

Figure 1.4: Effect of microalloying on recrystallization temperature [6]

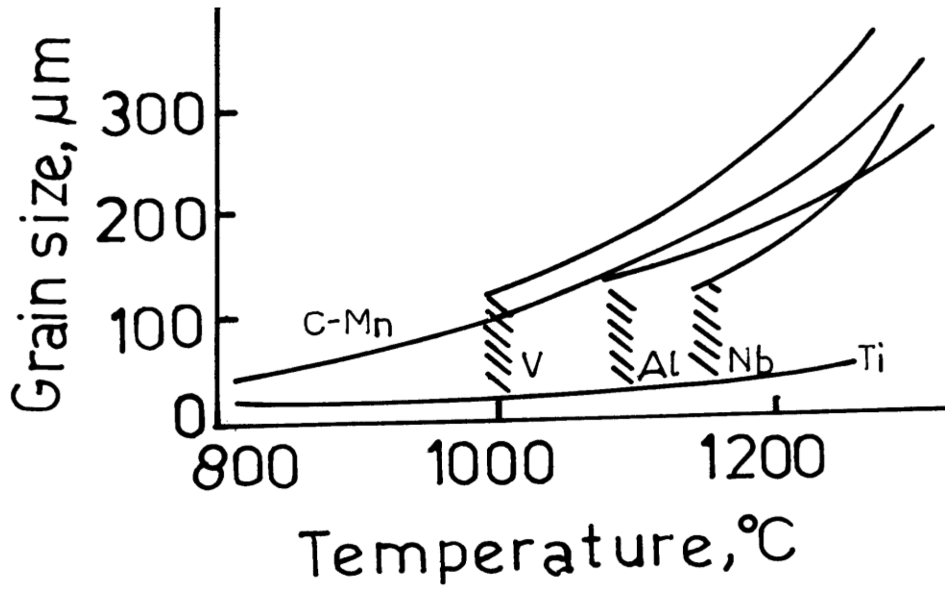

Figure 1.5: Effect of micro-alloying on austenite grain coarsening [5]

From figure 1.4 and figure 1.5, it can be observed that niobium is the most effective microalloying element in retarding recrystallization of hot worked austenite and only second to Titanium in controlling grain growth. The combined effect is such that niobium serves as the most important microalloying element in HSLA steel.

When a niobium bearing low alloy steel is in the austenite phase field, the niobium will be in the solid solution matrix and in the precipitated Nb(C,N), the partitioning depending on temperature. At equilibrium, this partitioning of niobium between the matrix and the precipitate will be controlled by the solubility relations [16].

The role of niobium as a microalloying element is summarised in figure 1.6.

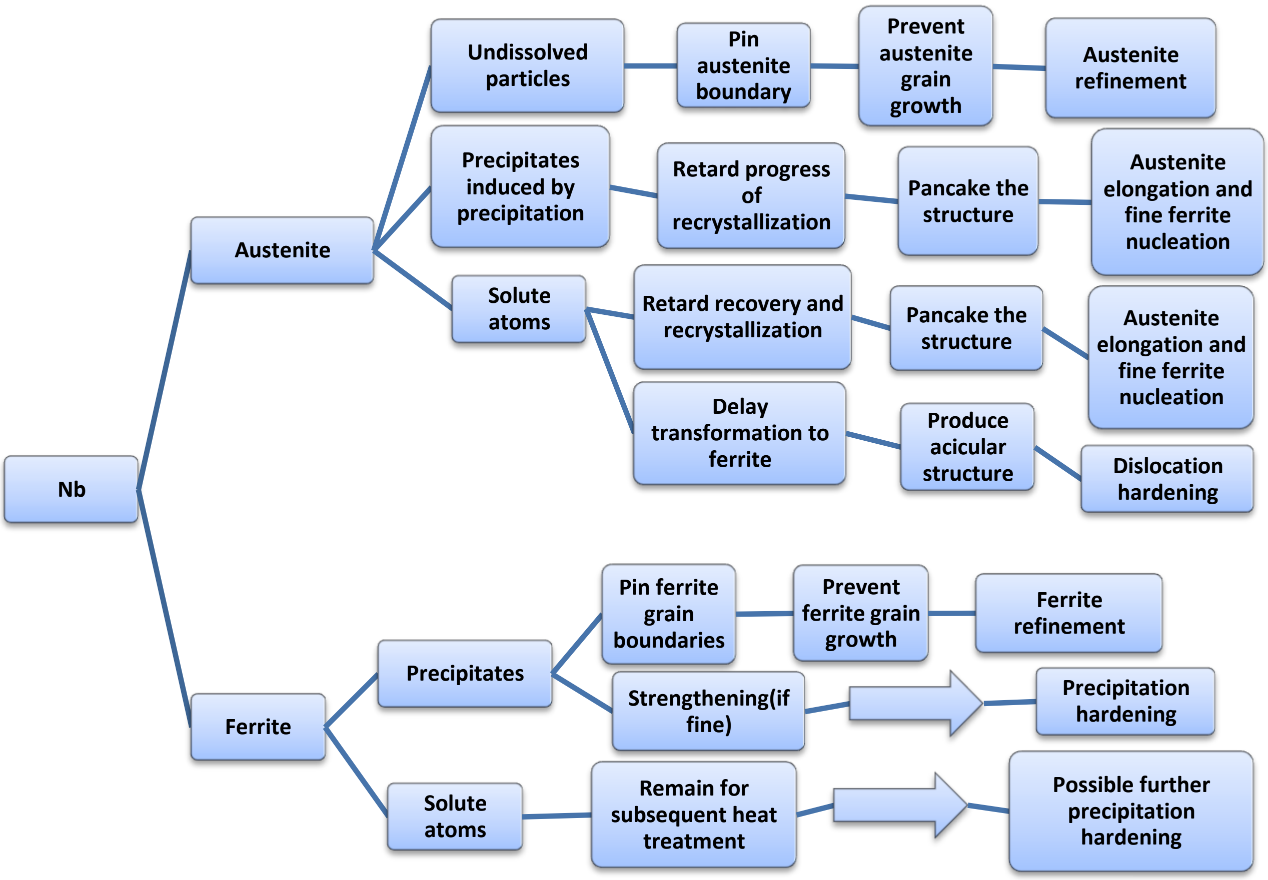

Figure 1.6: Role of Niobium as a microalloying element [10]

1.1.4 APPLICATION OF HSLA STEEL

In Russia, impressive constructions have been made from the newly developed construction steels and in total more than 450,000 tonnes of this high strength, low alloy steel have been used for bridges. New bridges have been built over many rivers including; the Ob in the city of Sirgut (Figure 3), the Don in Rostov-on-Don, the Oka in Kashira and the Moscow Canal in Dmitrov. Apart from this, HSLA steel also finds varied applications in the automotive industry ranging from several automotive profiles to Long member components like truck chasis, etc.

Figure 1.7: Bridge over Ob river in Russia

Figure 1.8: Truck chasis

Figure 1.9: Structural steel

Figure 1.10: Large diameter pipeline

CHAPTER 2

LITERATURE SURVEY

2. LITERATURE SURVEY

[1] Qingbo Yu, Zhaodong Wang, Xianghua Liu and Guodong Wang have studied the effect of niobium on yield stress and ferrite grain structure. Two low carbon steels were hot rolled by thermo-mechanical process (TMCP), and one contains niobium (0.013%), and another does not contain niobium. By the comparison of tested results, it was found that the yield strength of Nb steel is 55MPa higher than that of Nb free steel on an average. In addition, the ferrite grain of Nb steel is finer than that of Nb free steel under the same finishing temperature, cooling rate, and final temperature. However, there are no Nb(C,N) particles to precipitate from austenite and ferrite by the observation of transmission electron microscopy (TEM). It can be concluded by theoretical analysis that both the increase of strength and the refinement of ferrite grain of Nb steel result from solid solution Nb atoms.

[2] A.J.DeArdo reviewed the basic behaviour of niobium in a wide range of steels, including not only the traditional steels but also some of the newer versions. Particular emphasis has been placed on the basic metallurgical principles that apply to these steels, for it is the application of these principles that allows the composition-processing–microstructure–mechanical property relationships to be rationalised and exploited. The application of basic metallurgical principles has resulted in a predictive capability that has led to alterations in composition and processing for the purpose of producing steels with superior mechanical properties and improved overall performance.

[3] P.D.Hodgson and R.K.Gibbs has developed a mathematical model that predicts the final mechanical properties of hot rolled steels. It consists of sub-models for static and metadynamic recrystallization, grain growth and the transformed ferrite grain size. Each sub-model was characterised for a wide range of C-Mn and HSLA steels. The total microstructure model has been integrated into process models and evaluated using production data for plate, structural, bar and strip rolling. Results to date indicate that the accuracy of the model is excellent and is suitable for the evaluation of new steel grades and the development of optimised thermomechanical processing routes.

[4] Young-Kook Lee, Jin-Myung Hong, Chong-Sool Choi and Jae-Kon Lee studied the effects of niobium content and cooling rate on ferrite and bainite start temperatures (Ar3, Bs) and microstructural features have been studied in niobium bearing ultralow carbon microalloyed steels. The Ar3 and Bs temperatures decrease as niobium content or cooling rate is increased. The dependence of Ar3 on cooling rate is greater than that of Bs in all niobium contents. The bainitic ferrite laths become longer and narrower with increasing niobium content and cooling rate, and niobium also shows a tendency to decrease polygonal ferrite grain size.

[5] L.L. Teoh in his paper reviewed the role that the microalloying elements play in bar and rod mill processing, and how they influence the evolution of microstructure of austenite and ferrite during the thermomechanical rolling schedules. It also examines the future requirements which include a better understanding of fabricators and end-user requirements, computer aided system for analysing thermomechanical processing, innovative strategies in alloy design and thermomechanical treatments, and prediction of properties in the final products.

CHAPTER 3

EXPERIMENTAL PROCEDURE

3. EXPERIMENTAL PROCEDURE

3.1 SAMPLING SCHEME

The experimental strip samples were obtained from Hot Strip Mill, TATA STEEL (Jamshedpur unit).The chemical composition of the grades considered is shown in Table 3.1. The two grades, E-34 and SC-224 contain 0.012% and 0.029% Nb respectively. The composition of the steel grade used by Hodgson and Gibbs in is shown in Table 3.2. Different Nb compositions were taken in consideration to examine the effect of varying Nb composition on the final properties (in this case, hardness) of the steel. The entire strip was divided into three equal sections, namely edge, quarter and centre and samples were cut out from each of these three sections. The samples were then sized according to the dimensions shown in Fig 3.2. A total of four experimental solutionizing temperatures were selected i.e. 1100 °C, 1150 °C, 1200 °C and 1250 °C as shown in Table 3.3.

3.1.1 CHEMICAL COMPOSITION

Chemical compositions of the micro-alloyed steel grades procured from HSM, TATA Steel:

Table 3.1: Chemical composition of the grades used in the experiment

GRADE	C (%)	Mn (%)	S (%)	P (%)	Nb (%)	Ti (%)	V (%)	Si (%)	Al (%)	N_2 (ppm)
E-34	0.08	0.57	0.006	0.016	0.012	–	–	0.037	0.037	27
SC-224	0.08	1.39	.007	0.019	0.029	0.017	–	0.060	0.036	26

Table 3.2: Chemical composition of the unknown grade used by Hodgson and Gibbs

C (%)	Mn (%)	Nb (%)
0.18	1.3	0.035

3.1.2 SAMPLE LOCATIONS ON THE STRIP

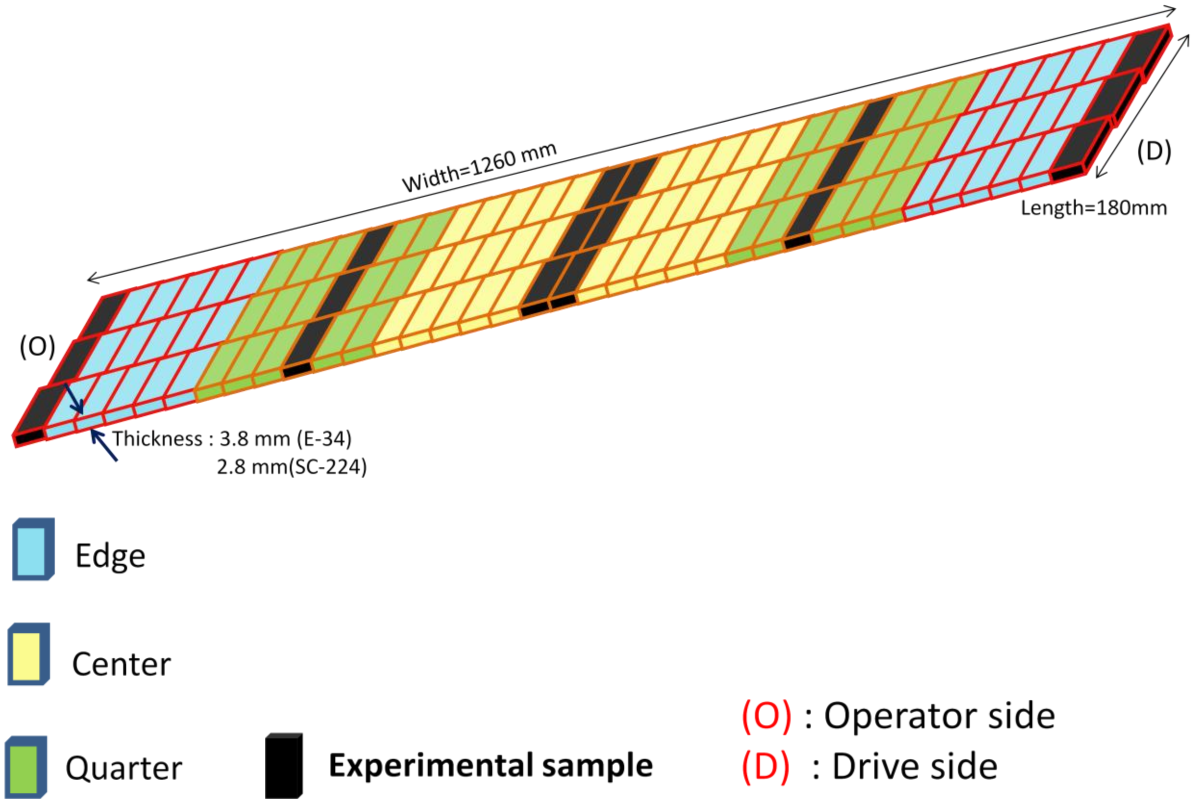

Figure 3.1: Sample location on strip

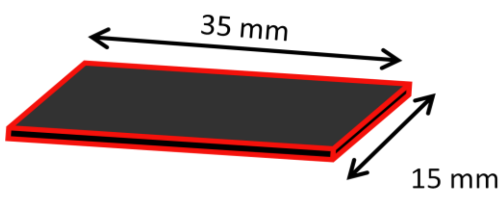

Figure 3.2: Sample dimensions

3.2 EXPERIMENTAL SOLUTIONIZING TEMPERATURES

Table 3.3: Experimental solutionizing temperatures and number of samples

		Experimental solutionizing temperatures °C			
		1100	1150	1200	1250
No of samples	Edge	3 (O)	3 (O)	3 (D)	3 (O)
	Quarter	3	3	3	3
	Centre	5	5	5	5

3.3 PROCEDURE

A qualitative estimate of the amount of Nb in solution was made by a series of quench and temper experiments. The samples were first heated (in air and nitrogen atmosphere separately) in a horizontal tube furnace (Kanthal) to the required solutionizing temperature, soaked at that temperature for half an hour, and then quenched in water at room temperature. Protective atmosphere (Nitrogen) was used to draw a comparison with heating in open (air) atmosphere, and characterization of the scales obtained when heating in air atmosphere was done to investigate the same. Post this process, the as-quenched hardness of the samples was recorded. The samples were then tempered at 575 °C in air atmosphere for half an hour and then allowed to cool to room temperature. Microhardness measurement was done using a Vickers hardness testing machine, and the obtained values were then plotted against the corresponding solutionizing temperatures. Further analysis was done to investigate the microstructural features and analyse the results obtained. Figure 3.3 represents the heating cycle employed.

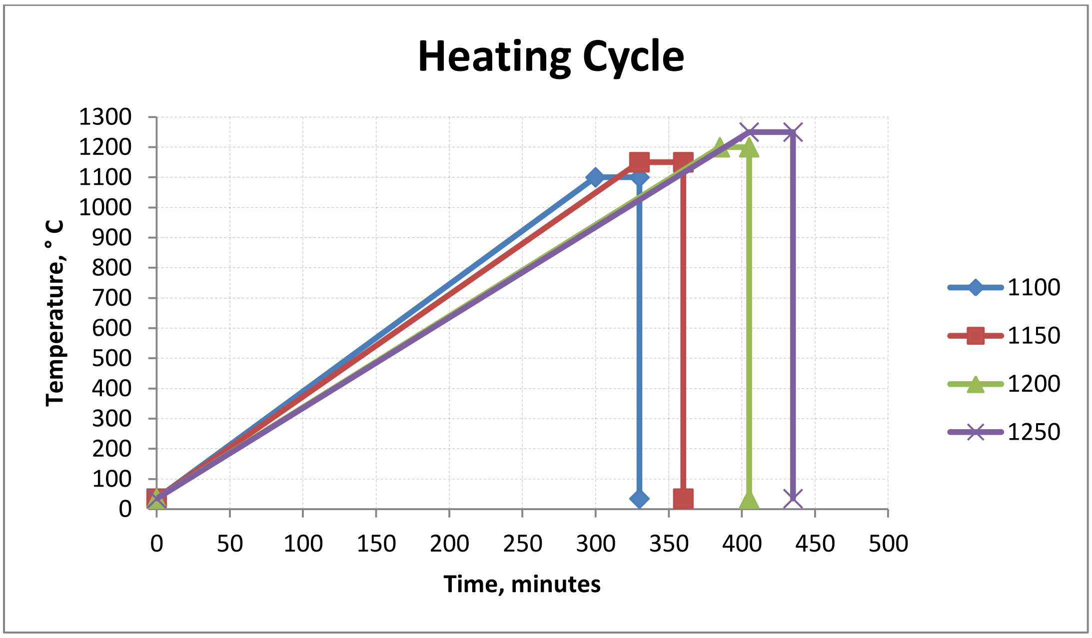

Figure 3.3: Representative heating cycle

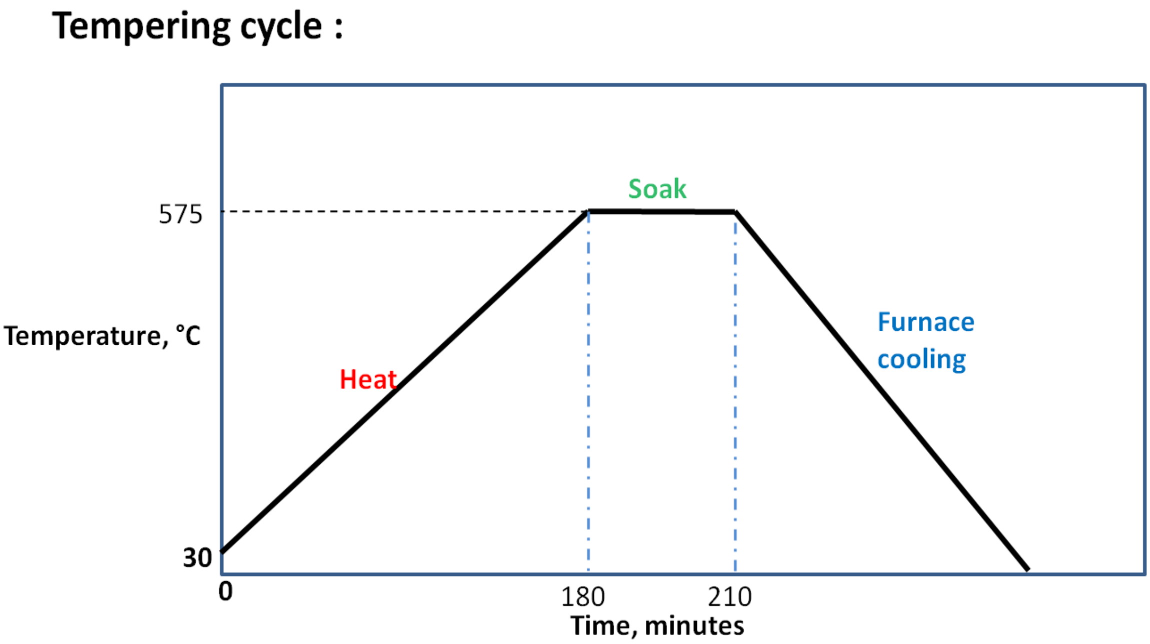

Figure 3.4: Representative tempering cycle

The as-tempered samples thus obtained were polished and hardness was recorded using a Vickers hardness testing machine.

CHAPTER 4

RESULTS AND DISCUSSION

4. RESULT AND DISCUSSION

4.1 EFFECT OF SOLUTINIZING TEMPERATURE ON AS-TEMPERED HARDNESS

The as-tempered hardness data was plotted with the corresponding solutionizing temperatures, and the following plot was established:

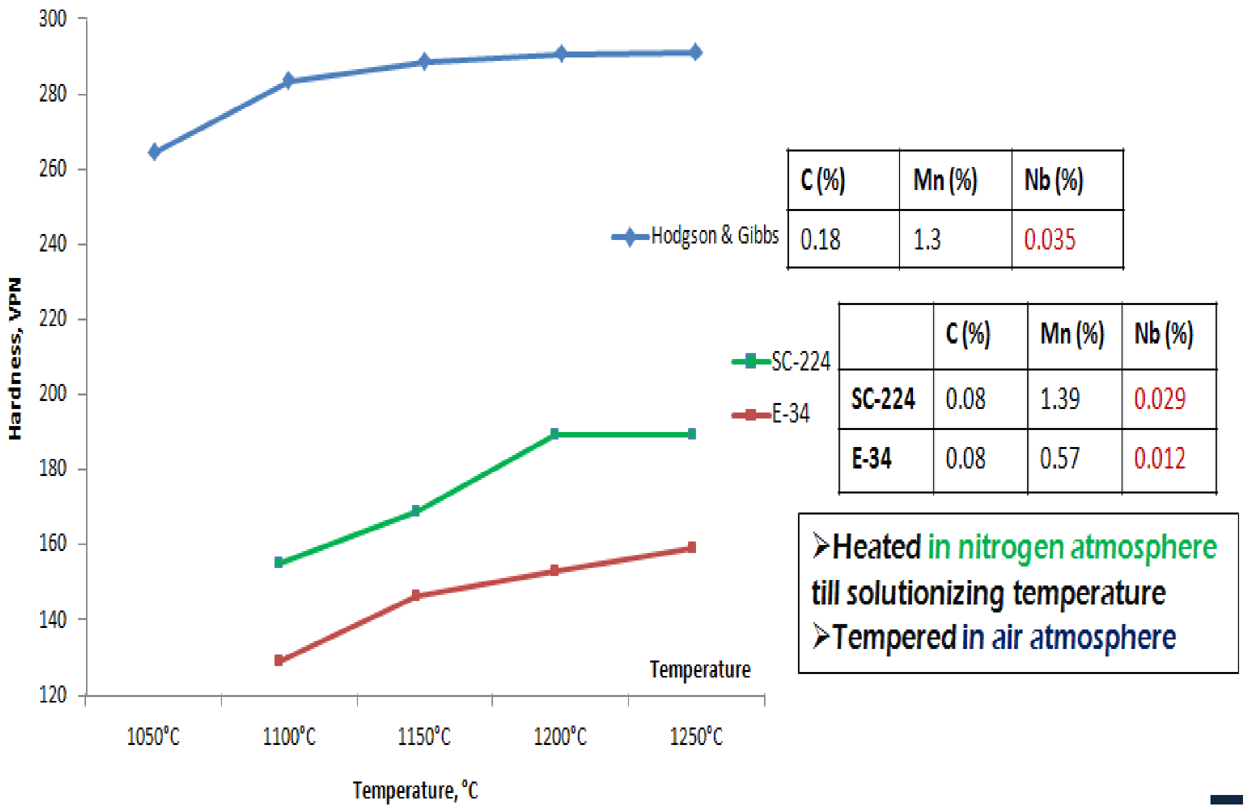

Figure 4.1: As-tempered hardness versus Solutionizing temperature plot for grades E-34, SC-224 and 'Hodgson and Gibbs' experiment

With increase in solutionizing temperature, the hardness increased abruptly at first (from 1100 °C to 1200 °C), and then gradually became almost constant in the range of 1200 °C to 1250 °C. The increase in hardness is attributed to the incremental amount of niobium that goes into the solution with the increase in solutionizing temperature. This added niobium that goes into the solution with every increment in solutionizing temperature finally precipitates as fine carbonitrides, Nb(C,N), during tempering operation, which imparts the observed increase in hardness.

Also, with an increase in the niobium content of the grade, the hardness value is observed to increase. This is because with increase in niobium content, the amount of niobium that goes into the solution increases. Consequently, the amount of carbide precipitating in the tempering stage also rises, and hence the increase in hardness.

Thus from figure 4.1, the following conclusions can be drawn:

- Nb(C,N) has dissolved and secondary hardening has occurred during tempering which lead to an increase in hardness .
- With increased dissolved Nb, the hardness increases. Hence the amount of dissolved niobium is a function of temperature.
- When Nb content in the steel is raised, the hardness increases for every solutionizing temperature due to increased fraction of dissolved Nb going into the solution which later precipitates during tempering.

Hence as observed, when temperature (for the composition range chosen, 0.012% and 0.029% Nb) is increased beyond 1200 °C, the increase in hardness as compared to the amount of heat input is considerably less. Therefore, heating beyond 1200 °C for complete dissolution on Nb(C,N) would be highly uneconomical and hence the solutionizing temperature range can be chosen to be between 1150 °C to 1200 °C for the grades considered.

4.2 HARDNESS VARIATION WITH SOLUTIONIZING TEMPERATURE ACROSS STRIP WIDTH

As discussed earlier, the entire strip (of both E-34 and SC-224 grade) was equally divided into three equal sections- edge, quarter and centre and samples from each of these sections was tested at the solutionizing temperatures considered (both air and nitrogen atmospheres).

The average as-tempered hardness of the edge, quarter and centre samples of each of the grades was plotted against the corresponding reheating temperature, and the following results were obtained.

<u>Grade E-34; Air atmosphere</u>

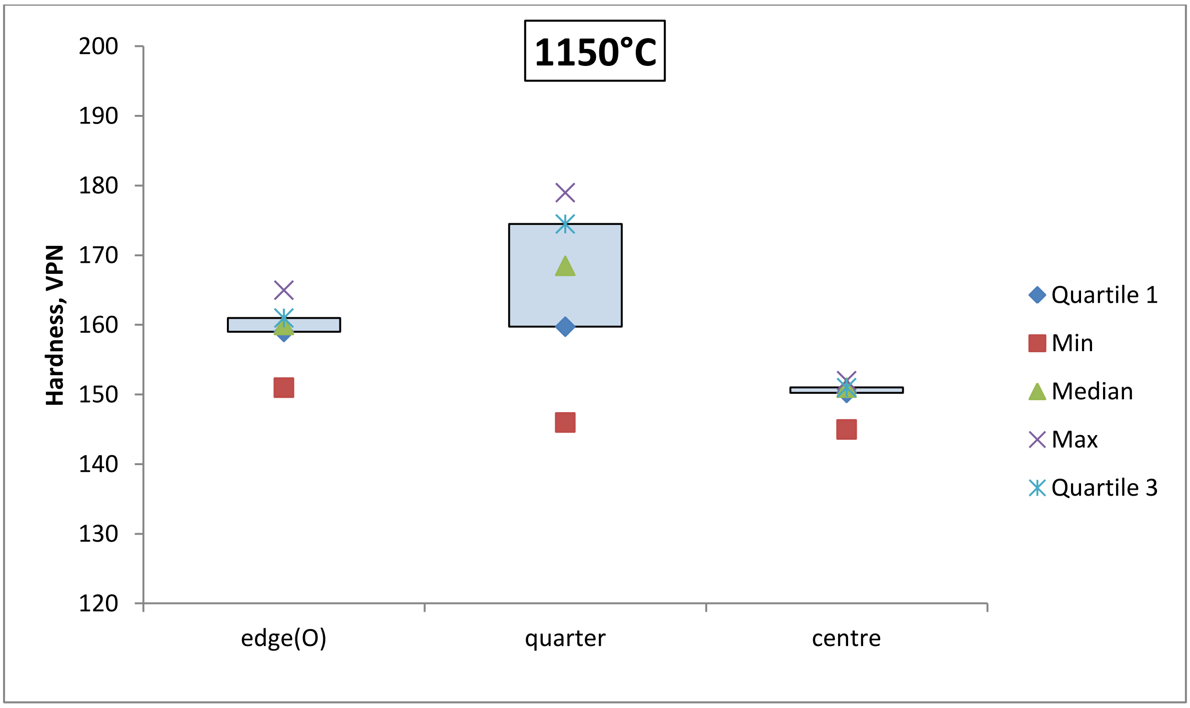

Figure 4.2: Hardness variation across strip width; Grade E-34; 1150 °C

Grade E-34; Air atmosphere

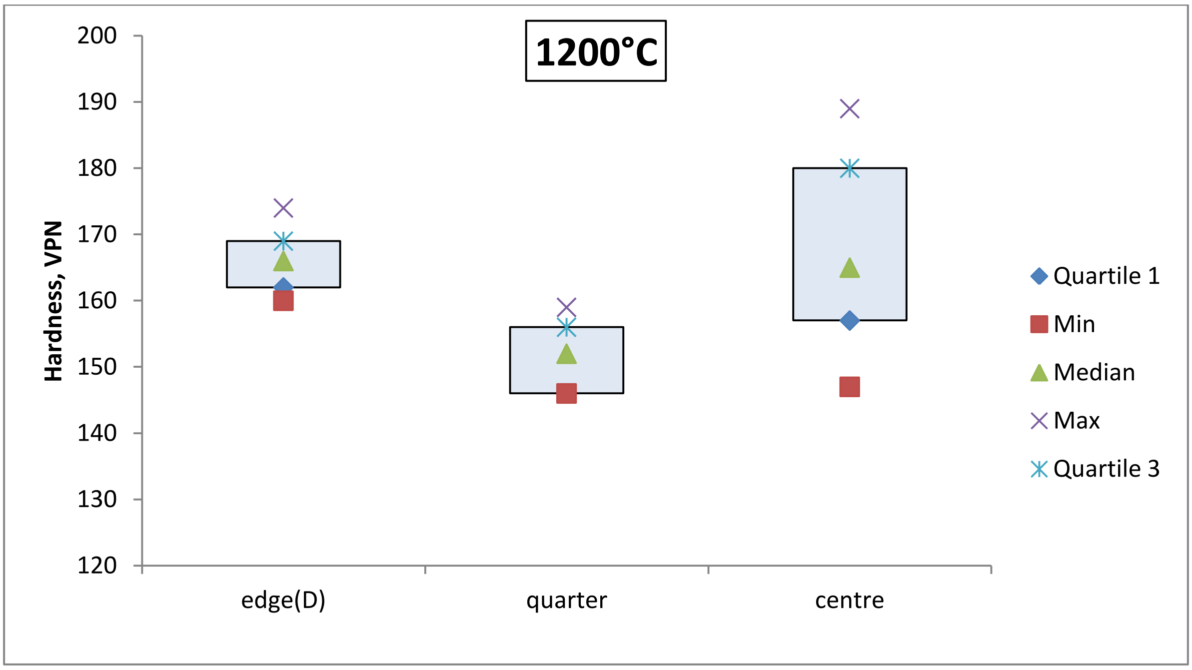

Figure 4.3: Hardness variation across strip width; Grade E-34; 1200 °C

Grade E-34; Air atmosphere

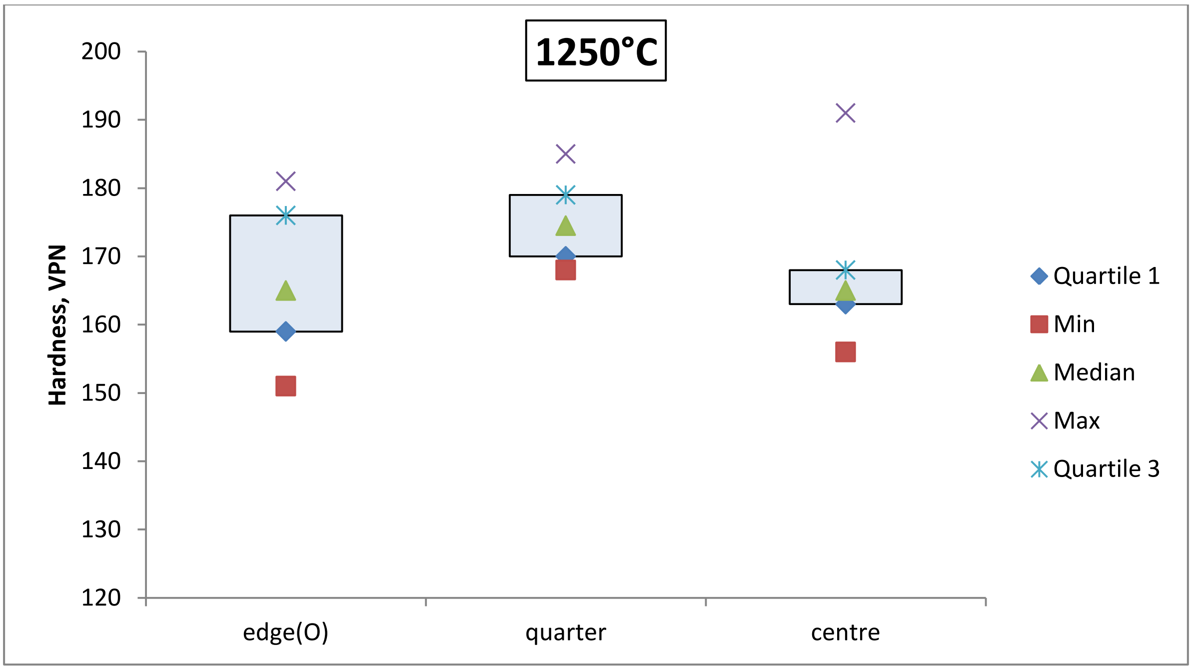

Figure 4.4: Hardness variation across strip width; Grade E-34; 1250 °C

Grade E-34; Nitrogen atmosphere

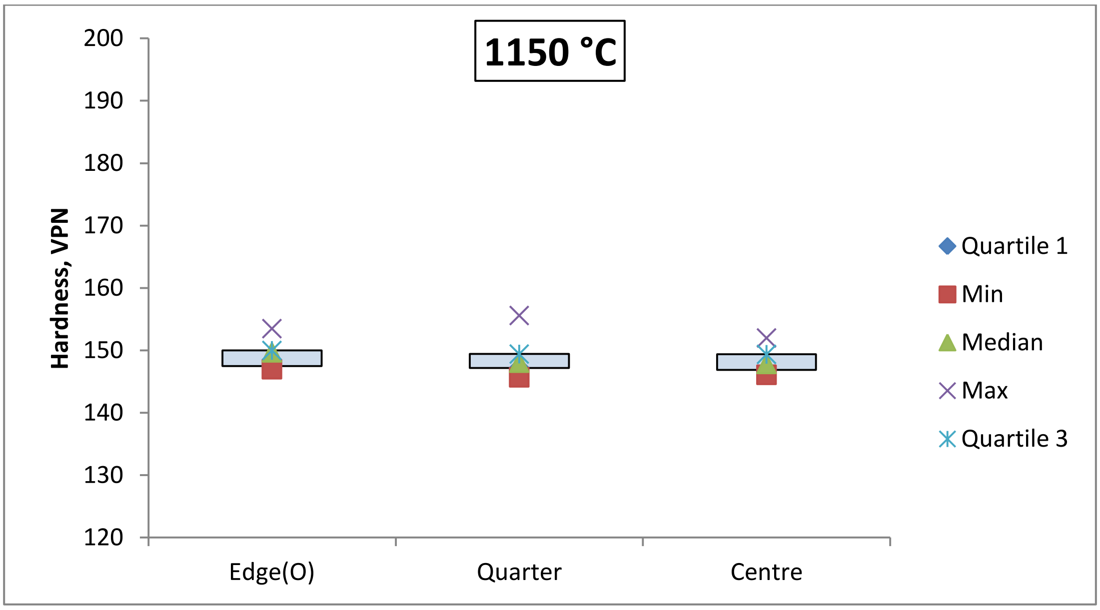

Figure 4.5: Hardness variation across strip width; Grade E-34; 1150 °C

Grade E-34; Nitrogen atmosphere

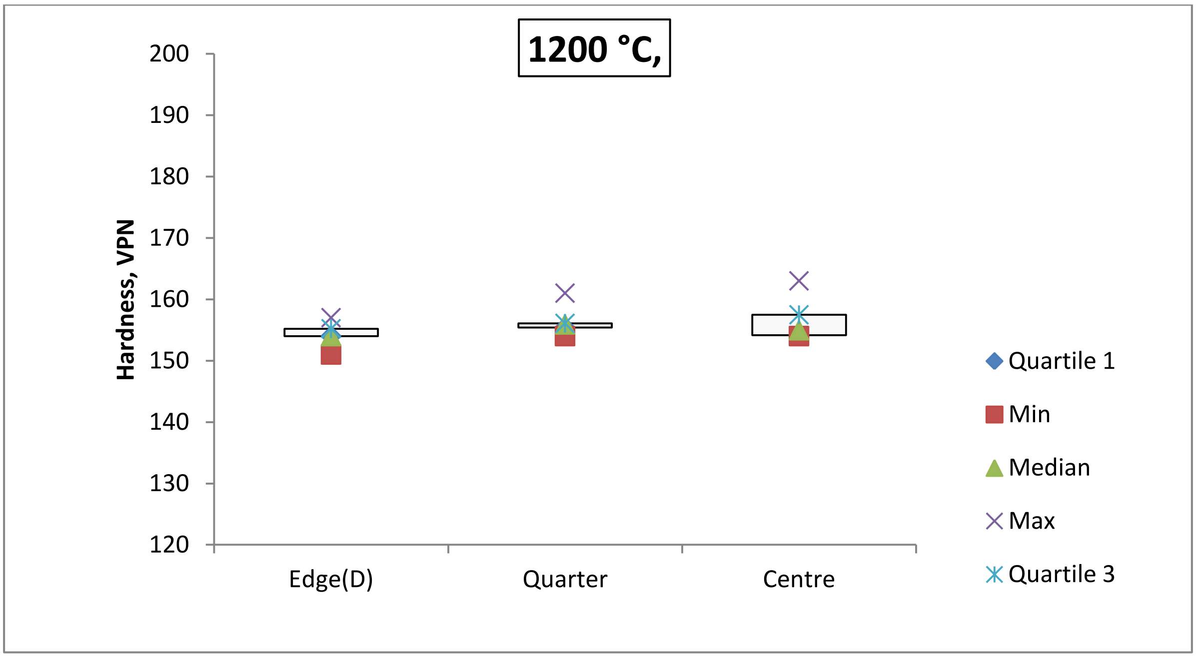

Figure 4.6: Hardness variation across strip width; Grade E-34; 1200 °C

Grade SC 224; Nitrogen atmosphere

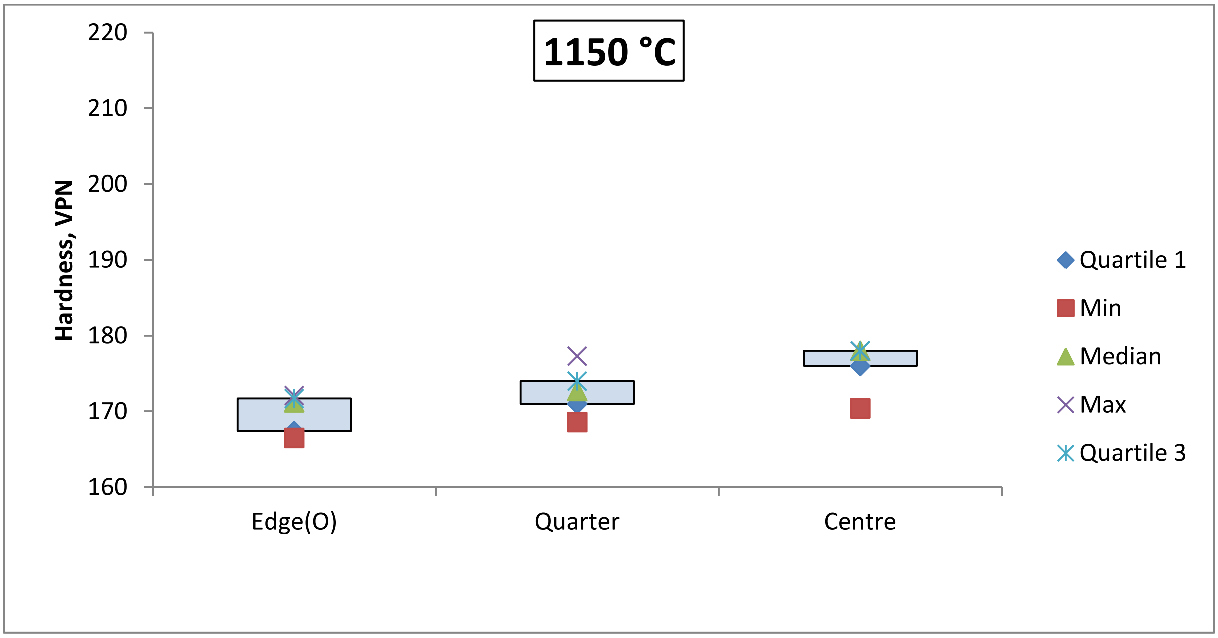

Figure 4.7: Hardness variation across strip width; Grade SC-224; 1150 °C

Grade SC 224; Nitrogen atmosphere

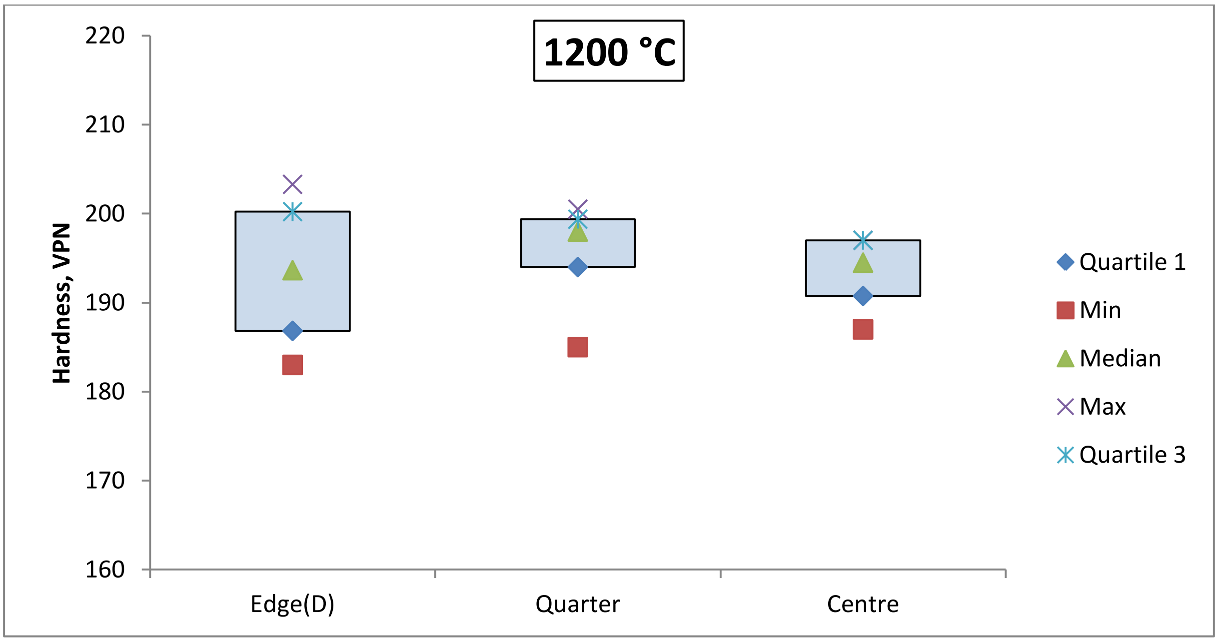

Figure 4.8: Hardness variation across strip width; Grade SC-224; 1200 °C

Grade SC 224; Nitrogen atmosphere

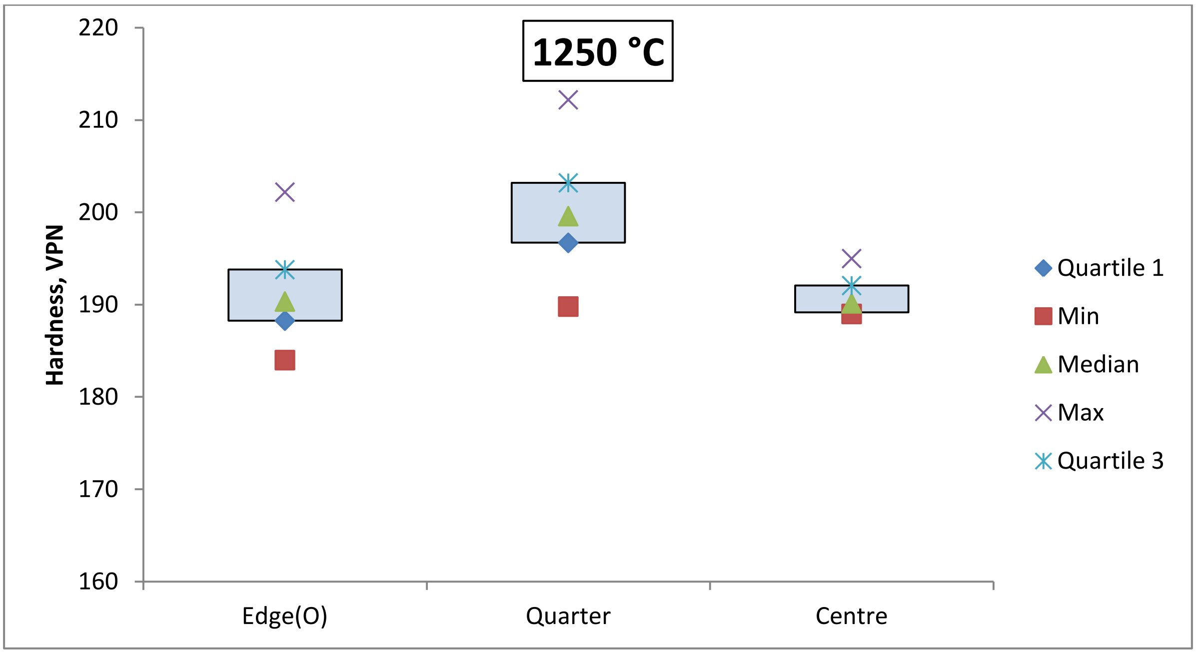

Figure 4.9: Hardness variation across strip width; Grade SC-224; 1250 °C

From the above plots, the hardness variation across the strip width at different solutionizing temperatures was found to be non-uniform for both the grades considered. In majority of the cases, the quarter samples exhibited a higher hardness value as compared to the edge and centre samples. Also, the magnitude of variation drastically reduced when heating was done in nitrogen atmosphere. The reason may be the absence of niobium depletion from the surface in the form of scales in case of heating in nitrogen atmosphere, which provided more uniformity in hardness values across the sample surface. For further investigation, an SEM characterization of the samples as well as that of the scales obtained (in case of heating in air atmosphere) were done to analyse the microstructure in an attempt to find a plausible solution to the observed phenomenon.

4.3 SCANNING ELECTRON MICROSCOPY

Microstructural analysis of the niobium containing steel grades post quenching and tempering was done under SEM (Scanning Electron Microscope) in an effort to observe the precipitation pattern of the niobium carbonitrides with solutionizing temperature. Due to limited resolution of the SEM, the exact nature of the carbonitrides could not be established. Also, further chemical analysis has been done with the help of EDS (Energy Dispersive Spectroscopy).

4.3.1 Grade E-34; 1250 °C; Quarter; Air atmosphere

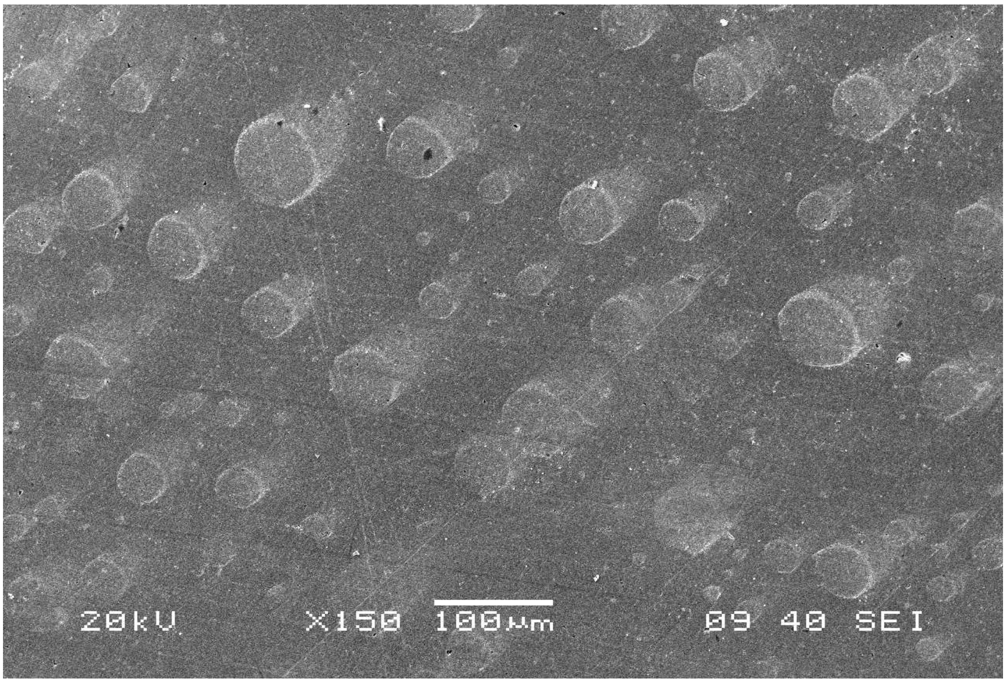

Figure 4.10: Scanned image of Grade E-34; 1250 °C; Quarter; Air atmosphere

The above image was viewed at an enhanced magnification to obtain more details about the interface that are visible in the image. It was suspected that niobium has segregated to the visible interfaces, thus rendering the sample with niobium depleted regions across the surface. Figure 4.11 shows a magnified image of the interface.

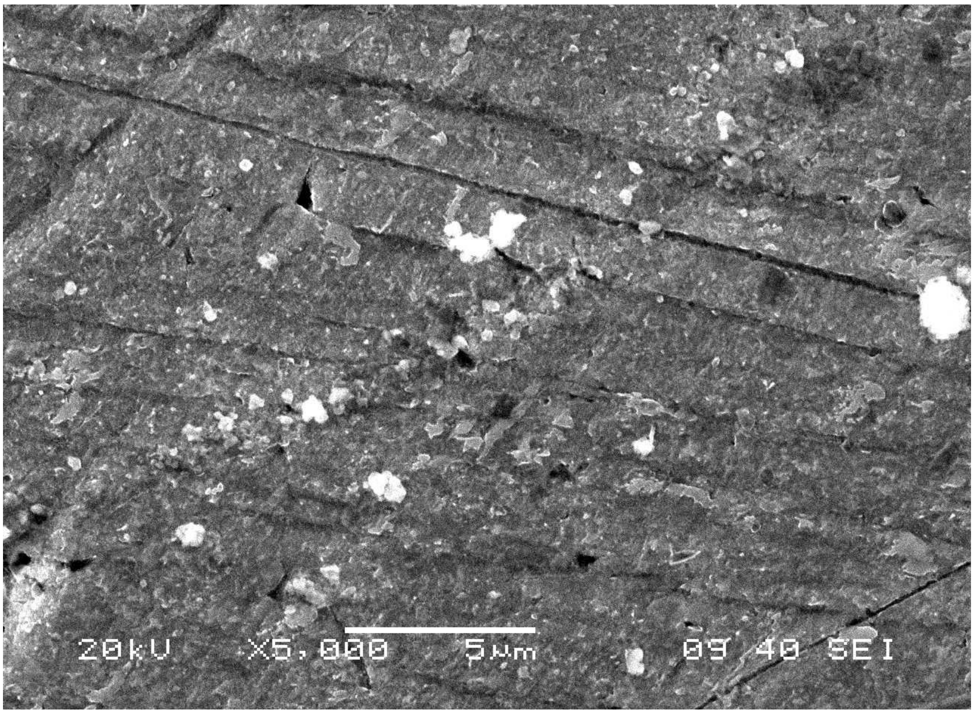

Figure 4.11: Scanned image of Grade E-34; 1250 °C; Quarter (interface); Air atmosphere

Further, EDS studies were done to verify the chemical identities of the gray and white patches visible in the image**.**

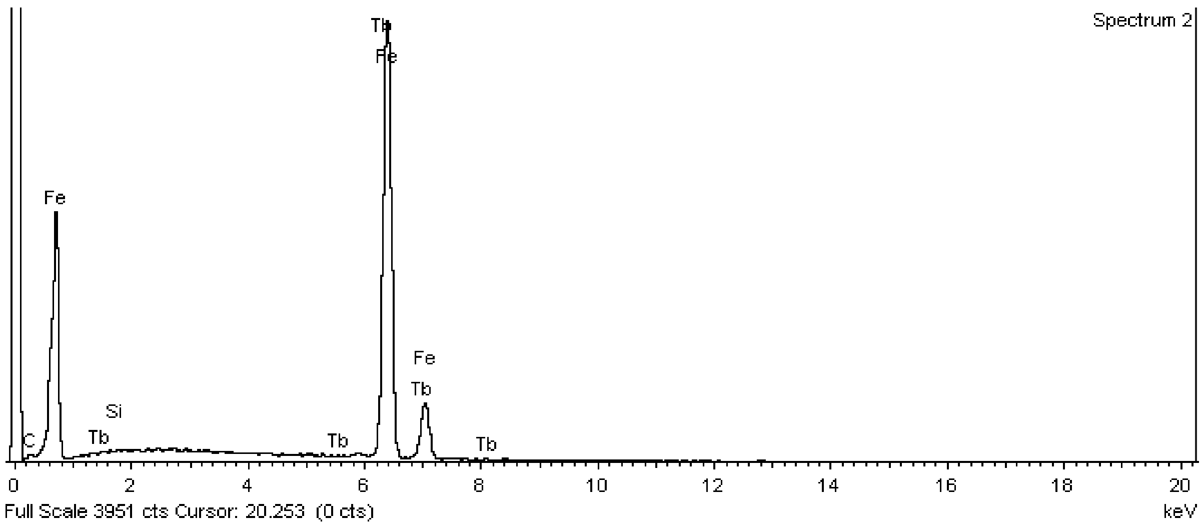

Figure 4.12: Scanned image of Grade E-34; 1250 °C; Quarter (gray part); Air atmosphere

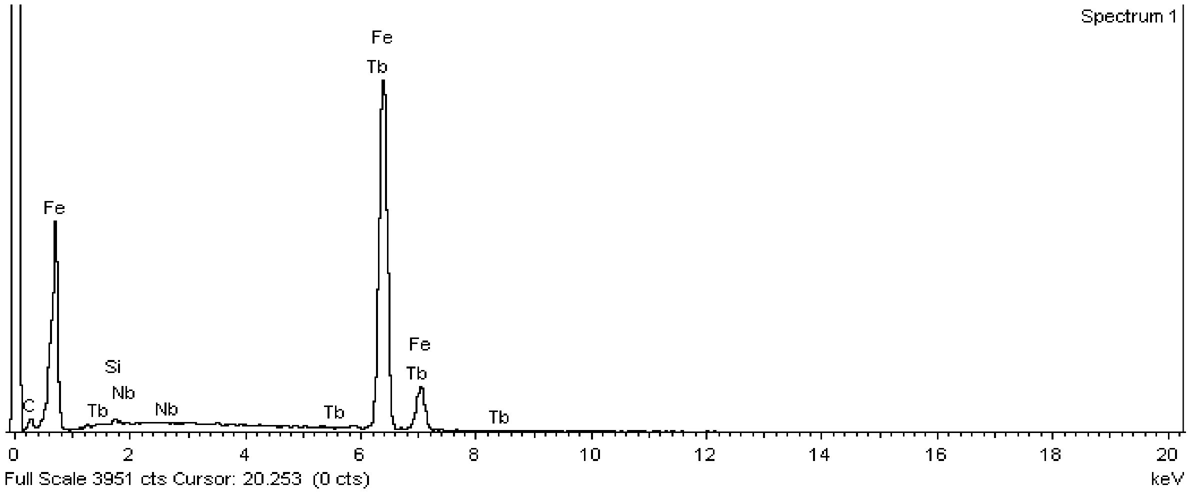

Figure 4.13: Scanned image of Grade E-34; 1250 °C; Quarter (white part); Air atmosphere

Hence, from the above EDS studies, it was confirmed that the white circular patches observed in the images were niobium segregated regions, whereas the gray patches were that of regions that have been depleted of niobium.

4.3.2 <u>Grade: E-34; 1150 °C; Edge; Air atmosphere</u>

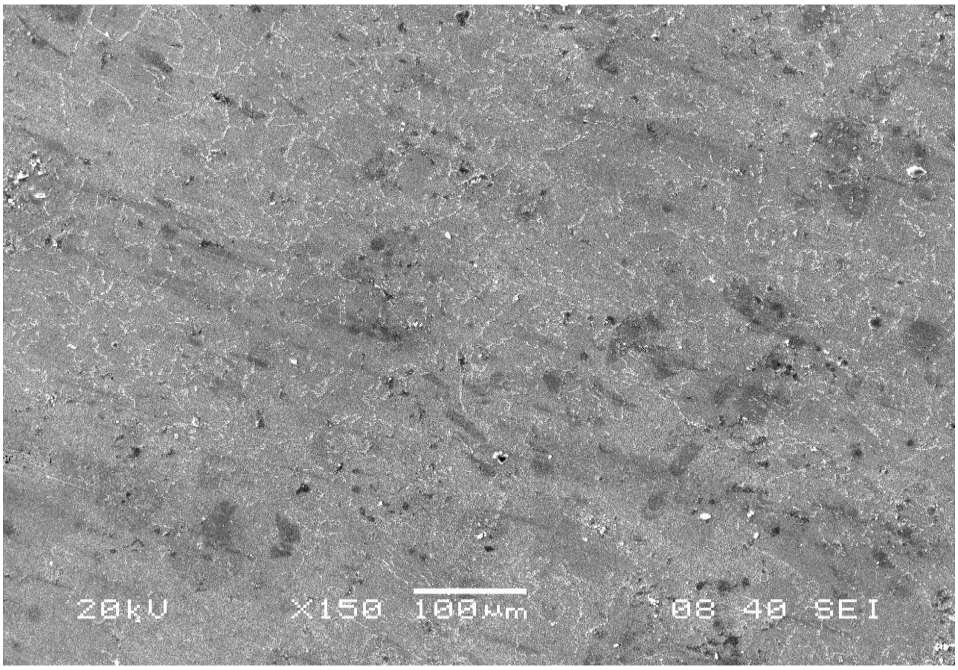

Figure 4.14: Scanned image of Grade E-34; 1150 °C; Edge; Air atmosphere

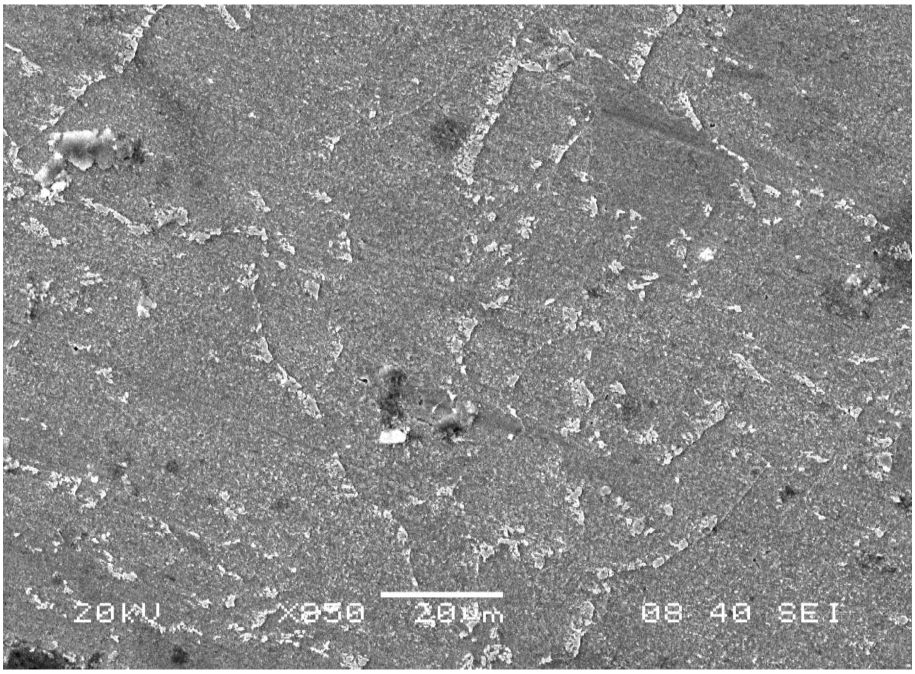

Figure 4.15: Scanned image (magnified) of Grade E-34; 1150 °C; Edge; Air atmosphere

4.3.3 Grade: E-34; 1150 °C; Quarter; Air atmosphere

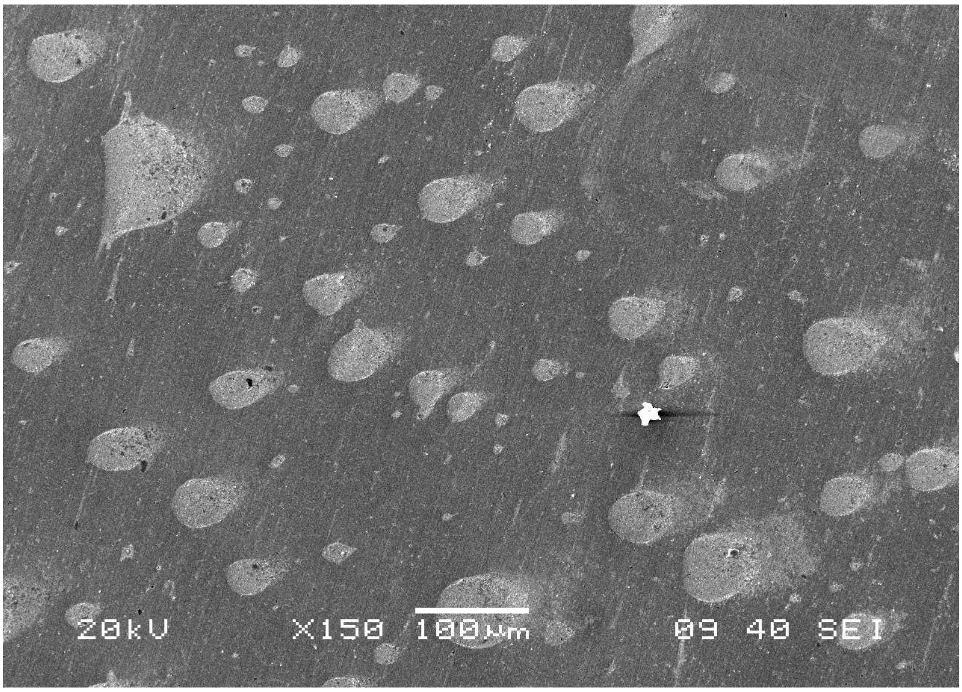

Figure 4.16: Scanned image of Grade E-34; 1150 °C; Quarter; Air atmosphere

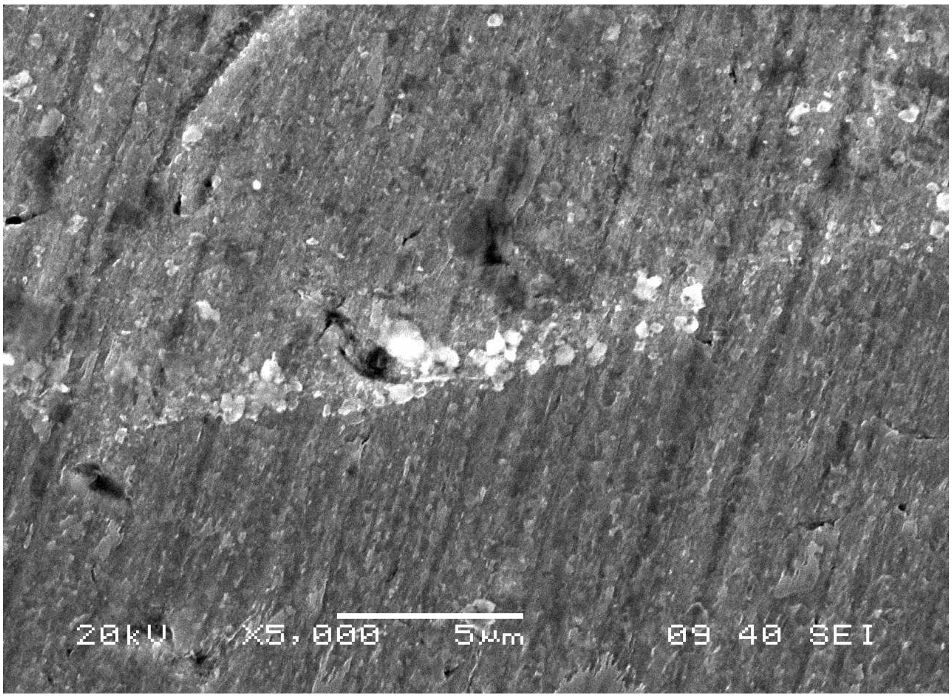

Figure 4.17: Scanned image (magnified) of Grade E-34; 1150 °C; Quarter; Air atmosphere

4.3.4 Grade: E-34; 1150 °C; Edge; Nitrogen atmosphere

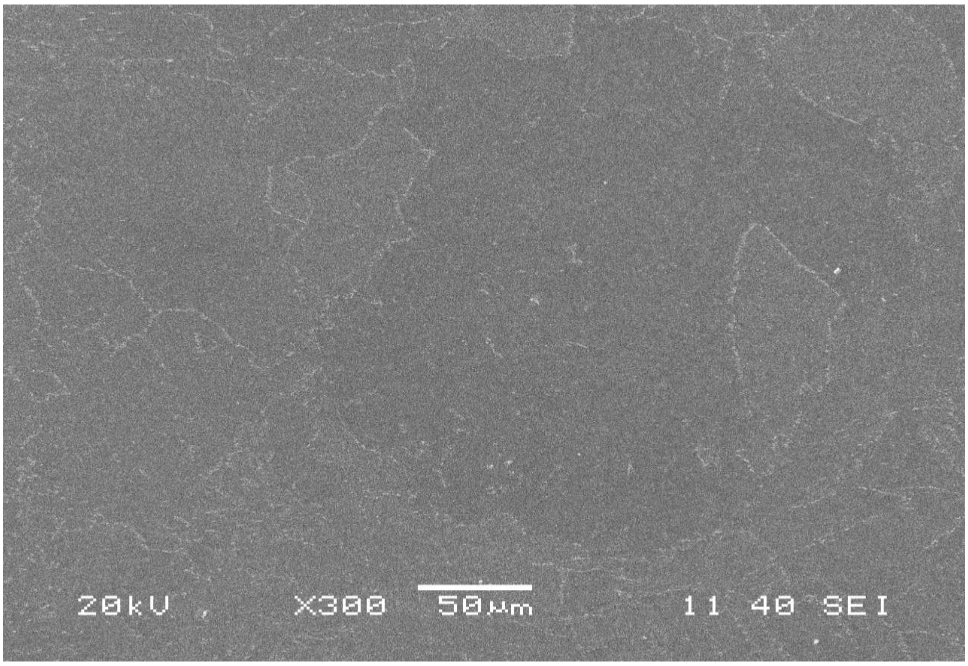

Figure 4.18: Scanned image of Grade E-34; 1150 °C; Edge; Nitrogen atmosphere

4.3.5 Grade: E-34; 1150 °C; Quarter; Nitrogen atmosphere

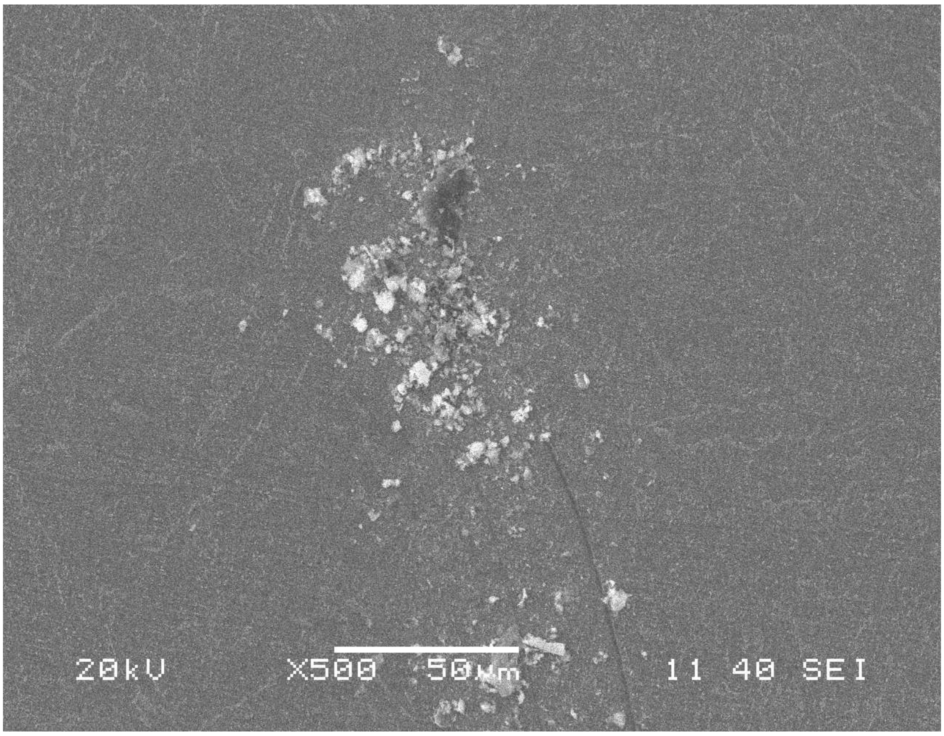

Figure 4.19: Scanned image of Grade E-34; 1150 °C; Quarter; Nitrogen atmosphere

4.4 SCALE CHARACTERIZATION

During heating in air atmosphere to the solutionizing temperature, it was observed that extensive scale formation has taken place. The thickness of the scale varied with the solutionizing temperature, a thicker scale being formed at a higher solutionizing temperature. Hence, the scale samples obtained for two temperatures- 1150 °C and 1250 °C were cold mounted across their cross-section and an SEM study along with EDS was carried out in an effort to characterize them, so that an approximate comparison can be drawn between heating in open (air) and protective (nitrogen) atmosphere. Scale formation was negligible in case of heating in nitrogen atmosphere.

4.4.1 <u>Grade E-34; 1150 °C; Quarter (scale); Air atmosphere</u>

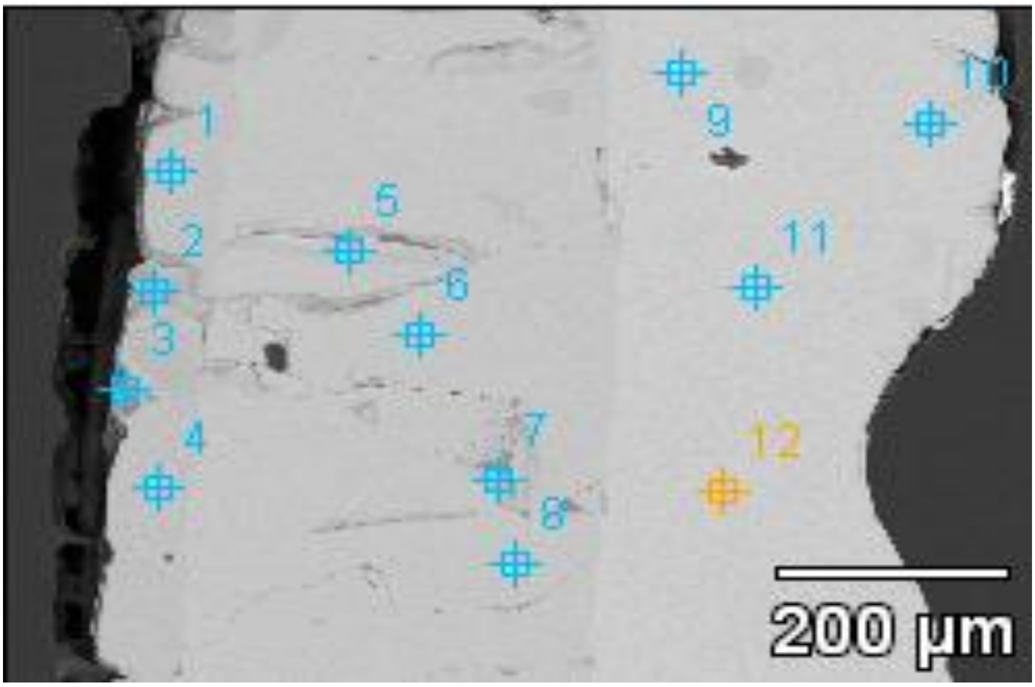

Figure 4.20: Scanned image of Grade E-34; 1150 °C; Quarter (scale); Air atmosphere

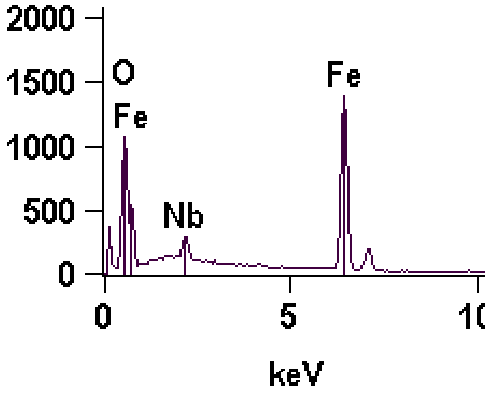

Figure 4.21: EDX pattern at point 1

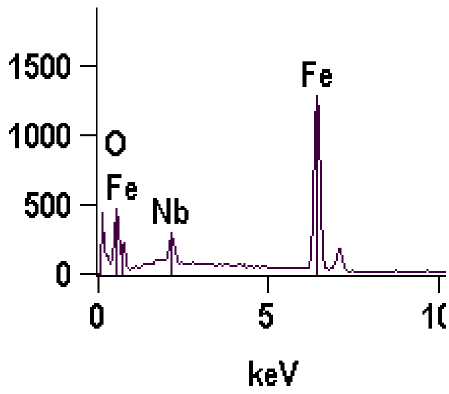

Figure 4.22: EDX pattern at point 2

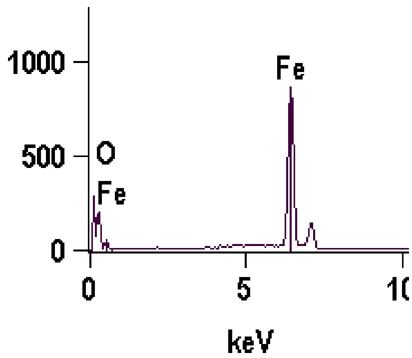

Figure 4.23: EDX pattern at point 3

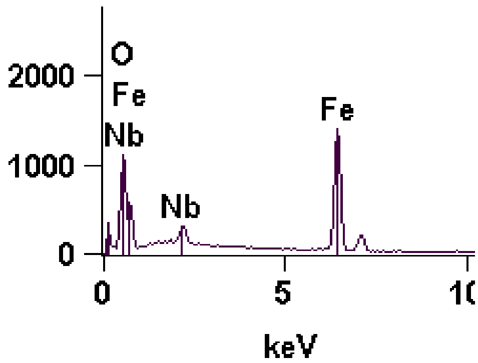

Figure 4.24: EDX pattern at point 4

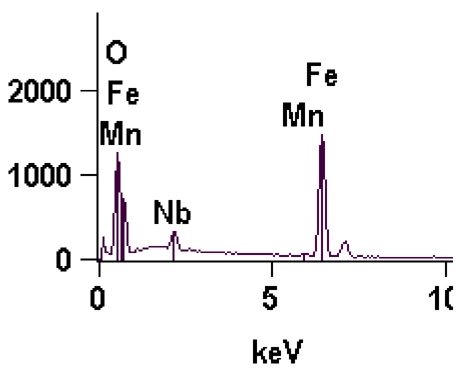

Figure 4.25: EDX pattern at point 5

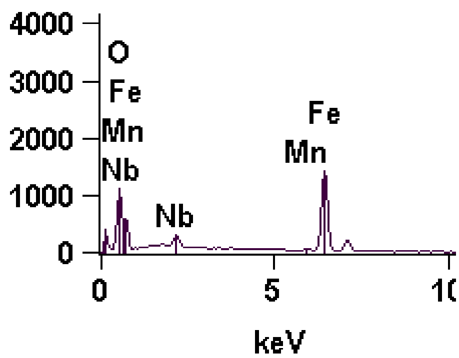

Figure 4.26: EDX pattern at point 6

4.4.2 Grade E-34; 1250 °C; Quarter (scale); Air atmosphere

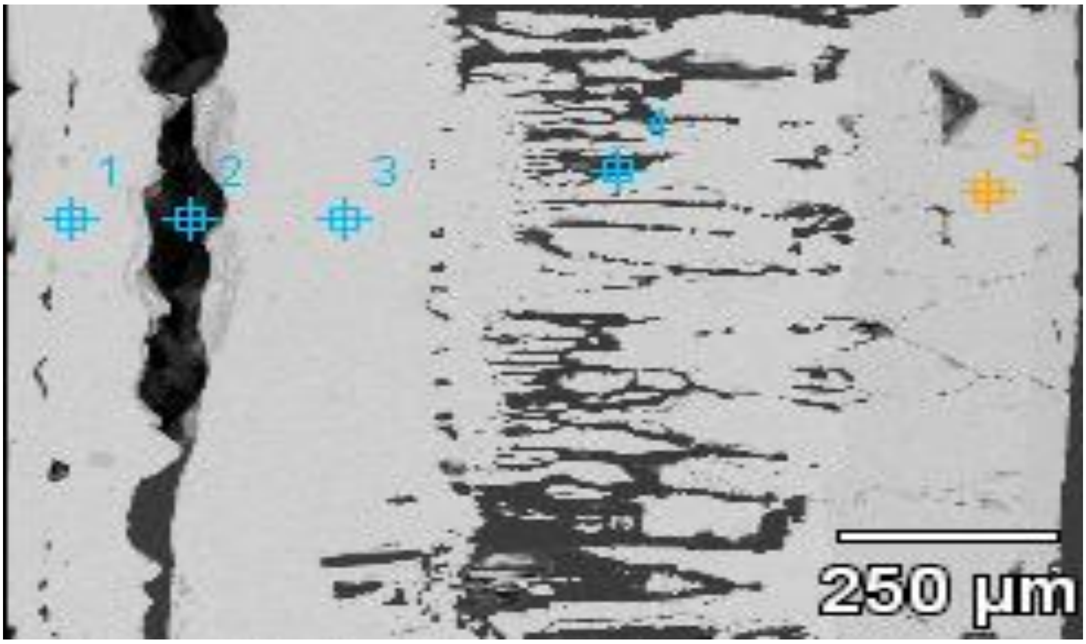

Figure 4.27: Scanned image of Grade E-34; 1250 °C; Quarter (scale); Air atmosphere

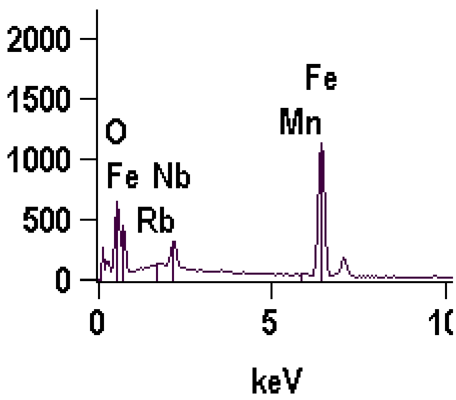

Figure 4.28: EDX pattern at point 1

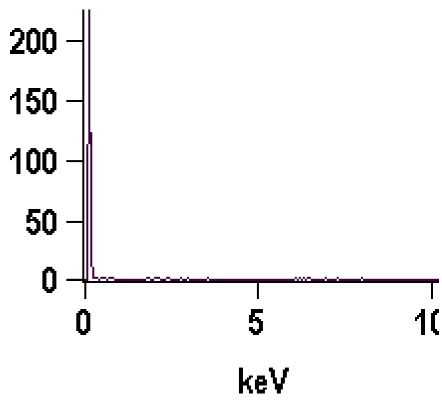

Figure 4.29: EDX pattern at point 2

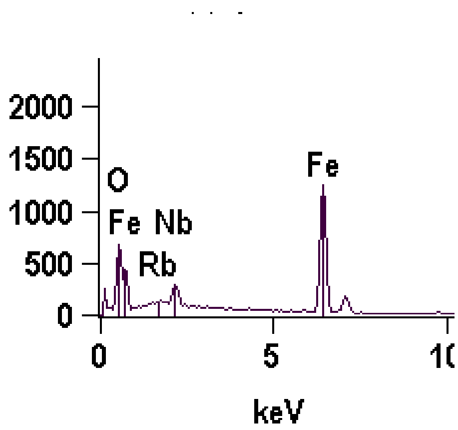

Figure 4.30: EDX pattern at point 3

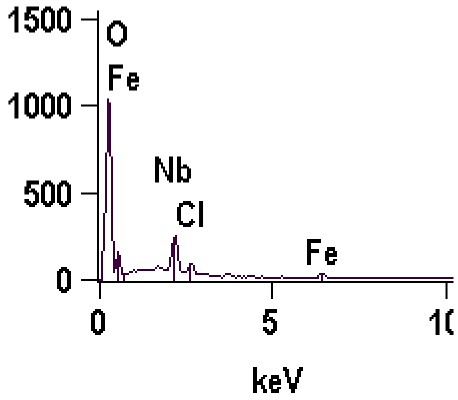

Figure 4.31: EDX pattern at point 4

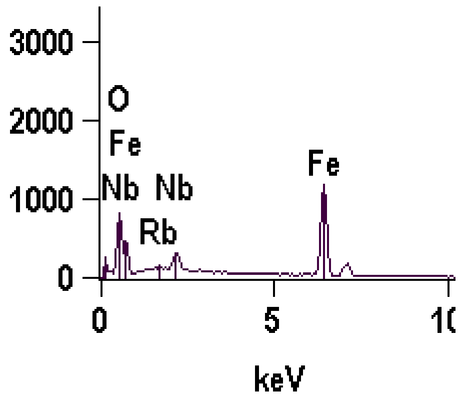

Figure 4.32: EDX pattern at point 5

From the above scanning micrographs and EDX analysis, it can be observed that there is considerable presence of niobium in the scales that were formed during heating to the solutionizing temperature in open (air) atmosphere. This is highly undesirable since niobium is an expensive constituent of microalloyed steel, and its loss in the form of scales would mean a reduction in the properties of the final product obtained.

CHAPTER 5

CONCLUSION

5. CONCLUSIONS

- Reheating temperature plays a critical role in determining the final properties of the HSLA steel, as it decides the initial grain structure where from the final microstructure is obtained

- Increasing microalloying content of the HSLA steel (Niobium in this case) leads to an increment in hardness, which is possibly due to
  - increased fraction of Niobium precipitates
  - additional refinement of grain structure

- Increasing the solutionizing temperature results in improvement of properties only up to a certain optimum temperature, after which Niobium levels in the solution saturate, and further heat input to raise the solutionizing temperature does not produce proportionate improvement in properties. Hence, it is advisable to reheat the steel within the optimum reheating temperature range.
- Hardness across strip width non-uniform, which may be due to segregation of Niobium compounds as suggested by SEM analysis

- Heating in controlled atmosphere can prevent the loss of Niobium as scales, which is both an expensive and the most important microalloying element in HSLA steel.

CHAPTER 6

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6. REFERENCES

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